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Crystal structure of methyl 2-((1-(2-(methoxycarbonyl)benzyl)-1*H*-1,2,3-triazol-4-yl)methoxy)-1-naphthoate, C₂₄H₂₁N₃O₅

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Abstract

C₂₄H₂₁N₃O₅, orthorhombic, *Pbca* (no. 61), *a* = 8.4167(7) Å, *b* = 14.0370(10) Å, *c* = 34.664(3) Å, *V* = 4095.4(6) Å³, *Z* = 8, *R*_{gt}(*F*) = 0.0457, *wR*_{ref}(*F*²) = 0.1001, *T* = 150(2) K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Methyl 2-(prop-2-yn-1-yloxy)-1-naphthoate (250 mg, 1.05 mmol) and methyl 2-(azidomethyl)benzoate (200 mg, 1.05 mmol) were added to *tert*-butanol: water = 8 mL: 8 mL in a 50 mL flask, then CuSO₄ · 5H₂O (50 mg, 0.21 mmol) and VcNa (100 mg, 0.52 mmol) were added. The mixture was stirred at room temperature for 15 h. The solution was diluted with water and extracted with ethyl acetate. The organic layer was dried over Na₂SO₄. After being filtered and concentrated,

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Table 1: Crystal collection and handling.

Crystal:	Block, colorless
Size:	0.320 × 0.290 × 0.120 mm
Wavelength:	Mo Kα radiation (λ = 0.71073 Å)
μ:	0.100 mm ^{−1}
Diffractometer, scan mode:	Rigaku Saturn 724 CCD, ϕ and ω-scans
θ _{max} , completeness:	25.0°, 100%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	21579, 3601, 0.0533
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2σ(<i>I</i> _{obs}), 2554
<i>N</i> (<i>param</i>) _{refined} :	291
Programs:	Bruker programs [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.51320(18)	0.93696(10)	0.33939(4)	0.0365(4)
O2	0.36574(18)	0.81591(10)	0.36067(4)	0.0371(4)
O3	0.55205(17)	0.50811(10)	0.39811(4)	0.0344(4)
O4	0.33380(16)	0.51874(9)	0.34033(4)	0.0309(4)
O5	0.49816(18)	0.50273(10)	0.28999(4)	0.0367(4)
N1	0.35294(19)	0.74176(11)	0.44629(4)	0.0247(4)
N2	0.4092(2)	0.73249(12)	0.48247(5)	0.0360(5)
N3	0.4973(2)	0.65466(12)	0.48293(5)	0.0344(4)
C1	0.5165(3)	0.89175(16)	0.30203(6)	0.0453(6)
H1A	0.581389	0.929776	0.284321	0.068
H1B	0.408001	0.886928	0.291932	0.068
H1C	0.562201	0.827825	0.304453	0.068
C2	0.4279(2)	0.89182(15)	0.36639(5)	0.0274(5)
C3	0.4191(2)	0.94782(14)	0.40299(5)	0.0238(5)
C4	0.4949(2)	1.03615(15)	0.40526(6)	0.0291(5)
H4	0.555444	1.058107	0.383906	0.035
C5	0.4838(2)	1.09244(15)	0.43798(6)	0.0310(5)
H5	0.537421	1.151950	0.439097	0.037
C6	0.3947(2)	1.06165(15)	0.46888(6)	0.0300(5)
H6	0.384625	1.100397	0.491217	0.036
C7	0.3197(2)	0.97368(14)	0.46721(6)	0.0288(5)
H7	0.259411	0.952612	0.488751	0.035
C8	0.3304(2)	0.91565(14)	0.43486(5)	0.0238(5)
C9	0.2446(2)	0.82078(14)	0.43689(6)	0.0275(5)
H9A	0.160331	0.824392	0.456752	0.033
H9B	0.193055	0.807876	0.411758	0.033

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C10	0.4024(2)	0.66959(13)	0.42383(5)	0.0232(5)
H10	0.378229	0.659055	0.397405	0.028
C11	0.4945(2)	0.61497(14)	0.44724(5)	0.0239(5)
C12	0.5779(2)	0.52400(14)	0.43830(5)	0.0260(5)
H12A	0.692841	0.529466	0.443924	0.031
H12B	0.533329	0.471037	0.453753	0.031
C13	0.5886(2)	0.42117(14)	0.38275(5)	0.0256(5)
C14	0.6789(2)	0.35339(14)	0.40330(6)	0.0305(5)
H14	0.709305	0.365288	0.429247	0.037
C15	0.7220(2)	0.27074(15)	0.38558(6)	0.0332(5)
H15	0.785107	0.226093	0.399358	0.040
C16	0.6760(2)	0.24937(15)	0.34740(6)	0.0298(5)
C17	0.5800(2)	0.31595(14)	0.32715(5)	0.0267(5)
C18	0.5396(2)	0.40414(14)	0.34538(5)	0.0249(5)
C19	0.5269(3)	0.29033(15)	0.28966(6)	0.0346(5)
H19	0.458115	0.331994	0.275984	0.042
C20	0.5734(3)	0.20661(16)	0.27298(6)	0.0422(6)
H20	0.536076	0.190834	0.247919	0.051
C21	0.6757(3)	0.14346(16)	0.29239(7)	0.0435(6)
H21	0.710424	0.086634	0.280124	0.052
C22	0.7244(3)	0.16428(15)	0.32876(7)	0.0403(6)
H22	0.792157	0.121058	0.341961	0.048
C23	0.4576(2)	0.47959(14)	0.32212(5)	0.0250(5)
C24	0.2563(3)	0.59621(15)	0.32001(6)	0.0358(5)
H24A	0.219097	0.573600	0.294833	0.054
H24B	0.331934	0.648504	0.316298	0.054
H24C	0.165584	0.618868	0.335163	0.054

the organic residue was purified by silica gel column chromatography, eluted with ethyl acetate and petroleum ether (1:1, v/v) to give a white solid (yield 68%, 310 mg); m.p. 84.9–85.7 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.02 (m, *J* = 8.0, 1H, Ar-*H*), 7.87 (d, *J* = 8.0, 1H, Ar-*H*), 7.79 (dd, *J* = 8.0, 1H, Ar-*H*), 7.73 (d, *J* = 12.0, 2H, Ar-*H*), 7.49 (ddd, *J* = 16.0, 1H, Ar-*H*), 7.39 (ddt, *J* = 24.0, 4H, Ar-*H*), 7.07 (dd, *J* = 8.0, 1H, Ar-*H*), 5.95 (s, 2H, CH₂), 5.41 (s, 2H, CH₂), 3.95 (s, 3H, CH₃), 3.89 (s, 3H, CH₃). Single crystals of the title compound were obtained from the solvents (ethyl acetate: petroleum ether = 1: 4).

Experimental details

All H-atoms were placed in calculated positions and treated as riding: C–H = 0.95–0.99 Å, with $U_{iso}(H) = 1.2$ or $1.5U_{eq}$ (parent C-atom).

Discussion

1,2,3-triazole is a five-membered heterocyclic aromatic compound, first reported by Michael in 1893 [4]. The 1,2,3-triazole has unique physical properties, and it is very stable in the

metabolic transformation, redox, acid and alkali environment and thermal conditions [5]. In recent years, 1,2,3-triazole derivatives promoted the development in medicine [6], in pesticides [7] and material research [8] as well as inorganic and organic synthesis [9].

The crystal structure shows that the triazole ring connects the benzyl and the naphthyl moiety. It contains one molecule per asymmetric unit and is held together by van der Waals forces. All intramolecular bond lengths are normal. The dihedral angle between the triazole ring and the naphthyl moiety is 8.25°. So the backbone is nearly coplanar. The bond lengths and angles are in the expected ranges. The molecular confirmation and the crystal packing is stabilized by weak intermolecular C–H···O and C–H···N hydrogen bonds.

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References

1. Bruker. APEX2 (Version 2.1.4), SAINT (Version 7.34A) and SADABS (Version 2004/1). Bruker AXS Inc., Madison, WI, USA (2005).
2. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Cryst.* **C71** (2015) 3–8.
3. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112–122.
4. Michael, A.: Über die Einwirkung von Diazobenzolimid auf Acetylcyclohexan-carbonsäure-methylester. *J. Prakt. Chem.* **48** (1893) 94–95.
5. Malow, M.; Wehrstedt, K. D.; Neuenfeld, S.: On the explosive properties of 1-benzotriazole and 1-1,2,3-triazole. *Tetrahedron Lett.* **48** (2007) 1233–1235.
6. Irastorza, A.; Aizpurua, J. M.; Correa, A.: Triazole-directed Pd catalyzed C(sp²)-H oxygenation of arenes and alkenes. *Org. Lett.* **18** (2016) 1080–1081.
7. Wei, J.; Chen, J.; Xu, J.; Cao, L.; Deng, H.; Sheng, W.; Zhang, H.; Cao, W.: Scope and regioselectivity of the 1,3-dipolar cycloaddition of azides with methyl 2-perfluoroalkynoates for an easy, metal-free route to perfluoroalkylated 1,2,3-triazoles. *J. Fluorine Chem.* **133** (2012) 146–154.
8. Astruc, D.; Liang, L.; Rapakousiou, A.; Ruiz, J.: Click dendrimers and triazole-related aspects: catalysts, mechanism, synthesis, and functions. A bridge between dendritic architectures and nanomaterials. *Acc. Chem. Res.* **45** (2012) 630–632.
9. Gonda, Z.; Lorincz, K.; Novak, Z.: Efficient synthesis of deuterated 1,2,3-triazoles. *Tetrahedron Lett.* **51** (2010) 6275–6277.